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Synthesis and Characterisation of Novel Yttrium Schiff Base Complexes of Subsituted Diamines

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Abstract: The present study involves synthesis of Schiff Base ligands of substituted Diamines followed by complexation and formation of coordination complexes with metal, Yttrium (I). The analytical techniques used for characterisation of Schiff bases and their complexes were: Elemental Analysis, Spectral Analysis by UV Visible spectroscopy, IR and H¹ NMR. The complexes were also characterized using Thermal Analysis (TGA). Newly synthesized complexes are proposed to be of the general type [Y(I)L(W)]Cl] (L= ligand (Schiff base) synthesized from condensation of 1-phenyl-3-methyl-4-R-5pyrazolone with two diamines, W= coordinated water where R= acetyl/benzoyl). Ligand: Metal stoichiometry was observed to be 1:1. All new complexes are proposed to have square pyramidal geometry; wherein coordinated water is at axial position.

Keywords: Yttrium, Schiff base ligands, Azomethine group, Square pyramidal geometry

I. INTRODUCTION

Schiff base ligands are synthesised by condensation of an aldehyde/ketone group with a primary amine. Schiff base ligands has revolutionized research in the field of coordination chemistry. Presence of Azomethine group ($-N=CR_2$) in coordination with metal imparts significant applications to complexes as catalyst, anti-microbial agents, anti-fungal, anti-inflammatory and several other therapeutic attributes. Ligands with azomethine group also serve as backbone for the synthesis of copious heterocyclic compounds [1]. Multidentate (N & O donor atoms) Schiff bases has been substantially incorporated into forming complexes with transition metals [2].

Pyrazolone (heterocyclic compounds) entity and its derivatives, represents an interesting template for biopharmaceutical chemistry. This has been attributed to their uncomplicated synthesis and effective bioactivities. The coordination chemistry involving Schiff base ligands derived from Pyrazolone has been widely studied [3].

First metal of second transition series Yttrium provides lot of scope in complex formulation with Schiff bases. There is significant similarity between Yttrium metal and lanthanides. This unique property of Yttrium makes it highly suitable for complexation with Ligands as it leads to impressive interface in research [4].

Kumar C R Vinod synthesized complexes of Yttrium ion with 4-N-(2'-furfurylidene) amino antipyrine and reported coordination through oxygen of the carbonyl and nitrogen of azomethine group. The metal ions of novel complexes were proposed to show 1:1 stoichiometry vis-vis ligand [5]. M K M Nair et al synthesized and conducted study of phytochemical properties of Y (III) and Ln (III) 18 membered Schiff Base complexes [6]. E. M. Broderick et al, synthesized complexes of Y(III), Ce (III) and Ce (IV) with ligands derived from imino-phosphorane group and an imine group. Complexes were characterized using several spectroscopic techniques including cyclic voltammetry [7]. Yttrium and lanthanum were complexed with novel binuclear Schiff base ligands. Complexes exhibited 2:1coordination with Schiff bases [8].

V. Patroniak formulated 15 and 16 membered Y(III) complexes $[YL2(H_2O)_2] Cl_3.H2O$ and $[YL1(H_2O)_2](ClO_4)_3.2H_2O$. These complexes were synthesized in 1:1 stoichiometry with N302 and N5 macrocyclic Schiff base [9].

Ferrocene Schiff base ligand was complexed with dimeric yttrium phenoxide by S. Deng and P L. Diaconescu. Yttrium ion in complexes was reported to be in three different oxidation states. Catalytic role of these complexes was observed for the ring opening polymerization of cyclic esters and epoxides. Study indicated that oxidation of metal ions reduced catalytic activity in cyclic ester ring and reverse trends were observed for epoxide polymerization [10].

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Ligand 1,10 phenanthroline was complexed with Yttrium ion in 3+ OS. The *In vitro* biological of novel complexes were carried out. Complexes exhibited significant antibacterial and antifungal activity [11].

In view of the facts mentioned above and as part of our efforts to discover new potentially useful complexes, the present research is focussed on the synthesis and characterisation of New Yttrium complexes, derived from novel Schiff base ligands (Condensation of Pyrazolone derivatives with diamines), coordinating through N and O atoms.

II. EXPERIMENTAL

Materials and Physical Measurements

AR grade chemicals and solvents were used in the present study. Elemental analysis (C, H, N & O) was performed on a Thermo-Finnigan CHNO analyser (SAIF-IIT). The FTIR spectra was recorded on Shimadzu IR-Prestige-21, spectrophotometer in the range of 350cm⁻¹-4000cm⁻¹(Ruia College, Mumbai). The UV-Visible spectral analysis was carried out using Shimadzu UV-2600 in the range of 190-800 nm.

Complexes and ligands were also characterized by ¹H- NMR [NMR (300 Mhz-NMR2), Instrument of University of Mumbai]. Magnetic susceptibility values were recorded at Sherwood Scientific Magnetic Susceptibility balance (Banasthali University Jaipur). Thermogravimetric Analysis of samples were done at, USAA, Model Diamond TG/DTA (Saif-IIT-B). Samples were heated from 30°C to 1020°C, at the rate of 10°C/min.

Synthesis of Schiff Base Ligands (L1-L4):

Synthesis of Schiff base was two step procedure. First was substitution of an acetyl or benzoyl group at, fourth position of 3-methyl-1-phenyl pyrazol-5one, by reported method [12,13]. Second part was condensation of carbonyl group of Pyrazol-5-one with two diamines. The ligands L1, L2, L3 & L4 were obtained by refluxing primary amine (4,4'-di amino diphenyl methane and 4,4'-di amino diphenyl ether) with 4-R-3-methyl-1-phenyl pyrazol-5-one (R= acetyl/benzoyl) in a 1:2 molar proportion, in ethanol on water bath (18-22 hrs). 2-3 drops of conc. HCl were added to facilitate the reaction. Progress of the reaction was observed on TLC. Upon cooling, the product separated out, which was filtered and then recrystallized from alcohol. The yield of the product was approx. 60%.

Preparation of Complexes

Ligand solution (DMF) and added drop wise to the equimolar yttrium chloride (YCl₃. $6H_2O$) solution (water). Reaction mixture was stirred till the solid complex precipitated (4-5hrs). Through out the reaction pH was maintained at 8.0 by adding 1N NaOH. Complex was filtered and air dried. (Yield 20-25%)

III. RESULT AND DISCUSSION

The four Novel ligands [14] 4',4''-bis [(4-acetyl-3-methyl-1-phenyl pyrazol-5-one) methyl imino] diamino diphenyl methane (L1), 4',4''-bis [(4-benzoyl-3-methyl-1-phenyl pyrazol-5-one) methyl imino] diamino diphenyl methane (L2), 4',4''-bis [(4-acetyl-3-methyl-1-phenyl pyrazol-5-one) methyl imino] diamino diphenyl ether (L3) and 4',4''-bis [(4-benzoyl-3-methyl-1-phenyl pyrazol-5-one) methyl imino] diamino diphenyl ether (L4) and their respective complexes with Y (I) were synthesized. Table 1 shows the elemental analysis of all the Y(I) complexes.

Infrared Analysis:

Complete comparison of the peaks of ligands L1, L2, L3 and L4, with their Palladium complexes is presented in Table2. The characteristic peaks of free ligands (symmetric and antisymmetric vibrations v_{OH}) were predominantly observed as broad bands at 3250-3000 cm⁻¹ and 2350-3000 cm⁻¹. Low values detected can be attributed to inter/intra molecular H-bonding [15, 16]. The v_{CH} aromatic peaks and v_{CH} aliphatic peaks were assigned bands from 2800-2940 cm⁻¹ and 2300-2390 cm⁻¹ respectively. Main functional group in Schiff base is an azomethine group. Distinct and strong bands were observed around 1610-1580 cm⁻¹. These peaks are distinctly ascribed to to $v_{C=N}$ (azomethine) and $v_{C=N}$ pyrazolone groups [17]. The ligands L1, L2, L3 & L4, also exhibited distinct bands for δ_{OH} . 1220-1250 cm⁻¹ and v_{C-O} phenolic 1360-1388 cm⁻¹ [14,15]. On coordination with ligands, spectral analysis of complexes indicated band shift in the range of 2-50 cm⁻¹. Distinct blue shift was recorded in $v_{C=N}$ (azomethine) peaks, signifying coordination between Y (I) ion and ligands [14]. δ_{H2O} 815-880 cm⁻¹ peaks were observed for coordinated water molecules in IR spectra of metal

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complexes. New bands with medium to weak intensities, appeared in the regions 540-550 cm⁻¹ and 440-430 cm⁻¹, supporting the formation of the Y-O(v_{M-O}) and Y-N(v_{M-N}) respectively [5, 18].

Thermal Analysis: TGA of all the complexes of yttrium was carried at the rate of 10° C/min and were heated from **30°C to 1020°C**. Weight loss observed between **80-120°C** corroborated the presence of two molecules of water of crystallisation. Except for complex 4, [YL4(H₂O)] CL.H₂O where weight loss in the temperature range 80-120°C indicated one molecule (**Table 3**). Complexes were proposed to have one coordinated water molecule due to the weight loss between 150- 250 °C. All the four complexes showed significant loss of weight due to one molecule of coordinated water. After 300 °C till 600 °C there was loss of about 60-68% which indicates the disintegration of the whole complex. The conclusions of TGA spectra were supported by presence of spectral peaks in IR. [14,15].

NMR Analysis: ¹H-NMR spectra of ligands and complexes was carried out in deuterated DMSO, as both ligands and complexes were insoluble in commonly used solvents. The ligands L1, L2, L3 & L4 showed signals for phenolic OH at δ 12.662-12.933ppm [19]. Peaks due to methylene(-CH₂-) group were seen in ligands L1 and L2 at δ 4.066-3.8ppm and on complexation methylene peaks underwent shift to δ (4.212-3.7382) ppm in complexes [YL1(H2O)]CL.2H₂O and [YL2(H2O)]CL.2H₂O [20]. In ligands L3 & L4, the methylene was replaced by -O-, so characteristic peak of methylene group was missing [14]. The ligands L2 and L4 showed extra peaks at δ 6.157-6.988ppm showing replacement of acetyl group by benzoyl groups. These aromatic protonic peaks underwent shift on complexes de protonation of the phenolic proton did not occur. This was concluded due to the presence of NMR signal around δ 12ppm- δ 13ppm in the NMR spectra. Detailed peak values are in Table 4.

UV-visible Analysis: Yttrium metal ion was proposed to have $5s^{1}4d^{1}$ configuration due to the presence of two unpaired electrons. This was due to calculated magnetic moment values of 1.6 to 1.8 BM. The visible region electronic spectra of yttrium complexes show three weak intensity, broad bands appearing in the regions 470nm to 535 nm, 590nm to 690 nm and 730nm to 790nm. In d¹ configuration weak field as well as strong field splitting is same, so the ground state has been assigned as ${}^{2}T_{2}g$ [21]. In square pyramidal geometry the degenerate d_{xy} , d_{yz} , d_{xz} orbitals further splits into two sets with d_{xy} rising in energy. Similarly, e_{g} orbitals splits into two energy states with d_{x2-y2} being the highest [22]. **Table 5**.

Conclusion: Four Novel complexes of Yttrium are proposed to be paramagnetic (1.64-1.8 B.M) with two free electrons. Y(III) as present in yttrium chloride is reduced to Y(I) after acceptance of two electrons. The presence of unpaired electrons also indicates yttrium complexes to be high spin. Various spectral studies indicates that each yttrium complex has one coordinated water molecule 1-2 molecules of water of crystallization. Ligands L1, L2, L3 & L4 coordinates through N of azomethine group and O of hydroxyl group of the Schiff base. All complexes of yttrium have been proposed to have square pyramidal geometry (Fig 1). Ligand's donor atoms 'N' and 'O', occupy four equatorial positions in the same plane and coordinated water molecule is proposed to be at axial position.



Fig. 1 Proposed structure of Yttrium (I) Schiff Base Complexes

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	R	Ζ
Complex 1	-CH ₃	-CH ₂ -
Complex 2	-C ₆ H ₅	-CH ₂ -
Complex 3	-CH ₃	-0-
Complex 4	-C ₆ H ₅	-0-
bla 1. Flamontal Analysis Data of V (I)	complay	vas fram

rable 1. Elemental Analysis Data of 1 (1) complexes from Schiff Dase Ligands (L1-L4)								
COMPLEXES	COLOUR	M.P.	μ_{EFF}	ELEMENTAL ANALYSIS				
MOLECULAR FORMULA		°C		EXPECTED (OBSERVED)				
(FORMULA WEIGHT)		(TGA)		С	Н	Ν	0	
[YL1(H ₂ O)]CL.2H ₂ O.	Light Pink/	280	1.64	57.52	4.919	10.8732	23.608	
C ₃₇ H ₃₈ N ₆ O ₅ YCL	beige			(53.09)	(4.88)	(9.970)	(21.51)	
772.5								
[YL2(H ₂ O)] CL.2H ₂ O.	Yellow	400	1.64	62.91	4.68	9.369	8.8	
C47H42N6O5YCL				(63.77)	(5.045)	(9.663)	(9.804)	
896.5								
[YL3(H ₂ O)]CL.2H ₂ O.	Yellow	280	1.64	55.77	4.906	10.846	12.395	
C ₃₆ H ₄₀ N ₆ O ₆ YCL				(56.133)	(5.43)	(10.876)	(14.566)	
774.5								
[YL4(H ₂ O)]CL.H ₂ O.	Yellow	420	1.8	62.67	4.316	9.54	9.085	
C46H38N6O5YCL				(63.306)	(4.6483)	(9.935)	(10.398)	
880.5								

Table 2
Key IR bands (cm ⁻¹) of Y (I) complexes from Schiff base Ligands (L1-L4).

LIGAND/ COMPLEX	U _{OH} CM ⁻¹	U _{C=N} CM ⁻¹ AZOME THINE	U _{C=N} CM ⁻¹ PYRAZ OLINE	U _{C-0} CM ⁻¹ PHENO LIC	Δ _{OH} CM ⁻¹	Δ _{H20} CM ⁻¹	U _{M-N} CM ⁻¹	U _{M-O} CM ⁻¹
L1	3027-3065	1618.35	1580	1384.95	1225.82			
[YL1(H ₂ O)]CL.2H ₂ O	3007.02- 3068	1628.95	1577.84	1385.91	1243.18	868.97	437.86	542.02
L2	3027.41- 3065.02	1628.95	1592.31	1384.95	1227.4			
[YL2(H ₂ O)]CL.2H ₂ O	3063	1630.88	1591.34	1392.66	1284.65	868.97	434.97	542.02
L3	3400-broad	1626.06	1540.23	1384.9	1250.89			
[YL3(H ₂ O)]CL.2H ₂ O	3063	1654.03	1536.37	1382.06	1248	868.97	441.23	553.59
(L4	3033.19	1630.88	1573.98	1388.81	1246.07			
[YL4(H ₂ O)]CL.H ₂ O	3033.19- 3062.13	1620.27	1536.37	1384.95		865.11	440.01	551.67



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Table 4-
Thermogravimetric Analysis Data of V(I) Schiff Base Complexes

Thermogravitation in 1 (1) Sentir Buse Complexes								
COMPLEX	MASS BY	WATER OF	BY	COORDI	BY			
	FORMULA	CRYSTALLI	CALCULATION	NATED	CALCULATION			
		ZATION	%AGE OF	WATER	%AGE OF			
		LOSS OF	WATER OF	LOSS OF	COORDINATED			
		MASS B/W	CRYSTALL	MASS B/W	WATER			
		80 ^o C -100 ^o C	IZATION	150°C -300°C	MOLECULES			
[YL1(H ₂ O)]CL.2H ₂ O	772.5	4.5%	4.66 %	2%	2.33 %			
[YL2(H ₂ O)] CL.2H ₂ O	896.5	4%	4.01%	2%	2.007%			
[YL3(H ₂ O)] CL.2H ₂ O	774.5	4.5%	4.62%	2%	2.32%			
[YL4(H ₂ O)] CL.H ₂ O	880.5	2%	2.04 %	2.3%	2.04%			

Table 5

 ^1H NMR (δ ppm) Data of Schiff Base ligands and their Respective Y(I) Complexes

LIGAND/COMPLEX	δ-CH ₃ PROTONS AT 3, 3' PPM	δ - CH ₂ - PPM	δ PHENYL PPM	δ PHENOLIC PROTON PPM
L1	2.501-3.358	4.066	7.136-8.014	12.933
[YL1(H ₂ O)]CL.2H ₂ O	1.447-2.496 -3.362	4.212	New peaks at 6.755, 6.784 & 6.988 Besides 7.017-8.022	12.662
L2	2.501-3.370	3.8-3.9	New peaks at 6.844,6.872&6.974 Besides 7.001-8.013	12.706
[YL2(H ₂ O)]CL.2H ₂ O	1.4338-3.3770	3.7382	6.8488, 6.8625, 6.9767, 6.9905 7.1631-8.0030	12.6964
L3	1.00-2.389-3.369		7.141-8.022 ppm	12.912
[YL3(H ₂ O)]CL.2H ₂ O	2.3688-3.4848		7.1414-8.3053	12.909
(L4	1.447-2.496-3.362		New peaks at 6.755,6.784&6.988 Besides 7.017-8.022	12.662
[YL4(H ₂ O)]CL.H ₂ O	1.4533-2.4969- 3.5119		6.7585, 6.7737 & 6.7619 7.0049 - 8.0094	12.6291





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Table 6

Electronic Spectral Data Banus of Schill Base nganus and their Respective Y(1) Complexes							
LIGAND/ COMPLEX	Λ ₁ N П*	Λ ₂ П П*	Λ ₄ D-D	Λ ₅ D-D	$egin{array}{c} \Lambda_6 \ \mathbf{D} extsf{-}\mathbf{D} \end{array}$		
L1	271.59	346.91 (shoulder at 381.73)					
[YL1(H ₂ O)]CL.2H ₂ O	261.63	312.30	$\begin{array}{c} 474.08 \\ d_{yz} \rightarrow d_{xy} \end{array}$	$590.04 \\ d_{xz} \rightarrow d_{z2}$	$\begin{array}{c} 740.38 \\ d_{xz} \rightarrow d_{x2} \\ g_2 \end{array}$		
L2	262.70	338.02					
[YL2(H ₂ O)]CL.2H ₂ O	261.83	337.30	$\begin{array}{c} 604.03 \\ d_{yz} \rightarrow d_{xy} \end{array}$	727.36 broad $d_{xz} \rightarrow d_{z2}$			
L3	289.36	349.79					
[YL3(H ₂ O)]CL.2H ₂ O	261.80	312.34	$\begin{array}{c} 498.65 \\ d_{yz} \rightarrow d_{xy} \end{array}$	$\begin{array}{c} 654.15\\ d_{xz} \rightarrow d_{z2} \end{array}$	$744.28 d_{xz} \rightarrow d_{x2} y2$		
L4	311.35	347.59(shoulder at 382.07)					
[YL4(H ₂ O)]CL.H ₂ O	267.91	338.17(shoulder at 353.41)	$535.32 \\ d_{yz} \rightarrow d_{xy}$	744.96 broad $d_{xz} \rightarrow d_{z2}$			

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